AES and REELS Study of Fe/Ni Alloys

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(Received September 30 1998; accepted January 13 1999)

We have studied Fe, Fe80Ni20, Fe50Ni50, Fe30Ni70 and Ni using Auger electron spectroscopy (AES) and reflected electron energy loss spectroscopy (REELS). The surface atomic composition of these alloys was estimated almost close to the bulk composition after the sputter-cleaning, and the Auger line shapes taken with the high energy resolution of 0.05% were well reproduced by the spectral synthesis. Whereas there were no significant changes on the AES spectra, the REELS spectra were quite different in the zero-loss region. In this region the REELS spectrum of Fe50Ni50 is quite similar to that of pure Ni. Since Fe50Ni50 has a fcc structure as well as Ni, we believe that the zero-loss line shapes are strongly affected not only by the chemical composition but also by the crystal structure in the case of Fe/Ni alloys.

1. Introduction

In order to clarify and solve problems of quantification, chemical effects and so on in AES and XPS measurements of a variety of metal materials, the Metal Materials Group (MMG) of Surface Analysis Society of Japan (SASJ) has focused on some metal alloys. In the second half of 1997, we have studied the Fe/Ni alloys using Auger electron spectroscopy (AES) and refrected electron energy loss spectroscopy (REELS)[1]. The aim of this study is to examine the spectral changes caused by the interaction between Fe and Ni.

2. Experimental

The experiments were performed in a FE-AES system of VG model MICROLAB 310-F[2]. AES and REELS measurements were carried out by 10kV and 1kV electron beam, respectively, from a Schottky field emission source. Electron detection was provided with the concentric hemispherical analyzer (CHA), which can be used to obtain high energy resolution AES spectra.

Five samples of Fe, Fe80Ni20, Fe50Ni50, Fe30Ni70 and Ni were mechanically polished and rinsed with acetone. For the surface cleaning, an ion sputtering with a rastered Ar ion beam of 3kV was used.

3. Results and Discussion

3.1. AES Measurements

Figure 1 (a) and (b) show the AES spectra of each sample obtained from the as-received surface and from the sputter-cleaned surface, respectively. These spectra were taken with the conventional energy resolution of 0.5%. As a function of Ni content we can see the change of the Auger line shapes in the energy range between 550 and 900eV, which are attributed to the LMM transitions of both Fe and Ni.

At first in order to estimate the surface composition, we tried following two methods: (1) quantification with relative sensitivity

Table 1. Atomic composition[%] estimated with RSF provided by VG.

Sample	C	O	Fe	Ni	Fe/(Fe+Ni)
	(a) as-received				
Fe	44.5	33.2	22.3	0.0	100.0%
Fe80Ni20	46.6	31.4	19.7	2.3	89.7%
Fe50Ni50	48.2	28.2	12.8	10.7	54.5%
Fe30Ni70	53.6	22.1	8.2	16.1	33.7%
Ni	60.8	15.8	0.0	23.3	0.0%
	(b) sputter-cleaned				
Fe	2.7	7.4	89.9	0.0	100.0%
Fe80Ni20	N.D.	7.4	77.6	15.0	83.8%
Fe50Ni50	3.2	8.9	47.7	40.3	54.2%
Fe30Ni70	8.7	7.7	28.6	55.0	34.2%
Ni	10.9	5.7	0.0	83.5	0.0%

RSF used here are 0.555 for C-KLL, 0.424 for O-KLL, 0.426 for Fe-LMV and 0.392 for Ni-LMV, respectively.

Table 2. Normalized intensity of Fe-LMV and Ni-LMV (K-value) obtained from this study.

Sample	Fe-LMV	Ni-LMV	Fe/(Fe+Ni)	Fe-LMV	Ni-LMV	Fe/(Fe+Ni)
	(a) as-received			(b) sputter-cleaned		
Fe	0.460	0.000	100.0%	1.000	0.000	100.0%
Fe80Ni20	0.429	0.062	87.4%	0.793	0.191	80.6%
Fe50Ni50	0.277	0.261	51.5%	0.492	0.496	49.8%
Fe30Ni70	0.166	0.391	29.8%	0.307	0.670	31.4%
Ni	0.000	0.625	0.0%	0.000	1.000	0.0%

factors (RSF) provided by VG; (2) evaluation of K-values obtained from this study. Here we use the K-value as the normalized intensity of Fe-LMV or Ni-LMV, where the intensity of each peak is calculated from the integrated peak area of Fe-LMV (from 617 to 659eV) or Ni-LMV (from 741 to 797.5eV) after the background subtraction by Shirley method.

The results obtained are summarized in table 1 and 2. In each case, the atomic ratios of Fe/(Fe+Ni) after the sputter-cleaning were almost close to the bulk composition. This means that significant changes of the surface composition caused by such as the preferential sputtering do not occur on the Fe/Ni alloys in this study.

On the contrary, it seems that Fe is enriched to the as-received surface, as previously reported[3]. The composition of oxygen was likely to increase gradually according to the increase of Fe content. It could be considered that the preferential oxidation of Fe led to the segregation of Fe oxide to the Fe/Ni alloy surface[3].

Secondly we examined the AES spectral changes of both LMM and MVV transitions among Fe, Ni and Fe50Ni50. Figure 2 (a) and (b) show the AES spectra of Fe, Ni and Fe50Ni50 in the region of LMM and MVV transitions, respectively, which were obtained from the sputter-cleaned surface with the high energy resolution of 0.05%. We can observe that the AES line shapes are finer than fig. 1 by the improvement in energy resolution. Here we show the comparison of Fe50Ni50 spectra between the measured and the synthesized in fig. 3. The synthesized spectrum of Fe50Ni50 was made from the pure Fe and Ni spectra shown in fig. 2 in the

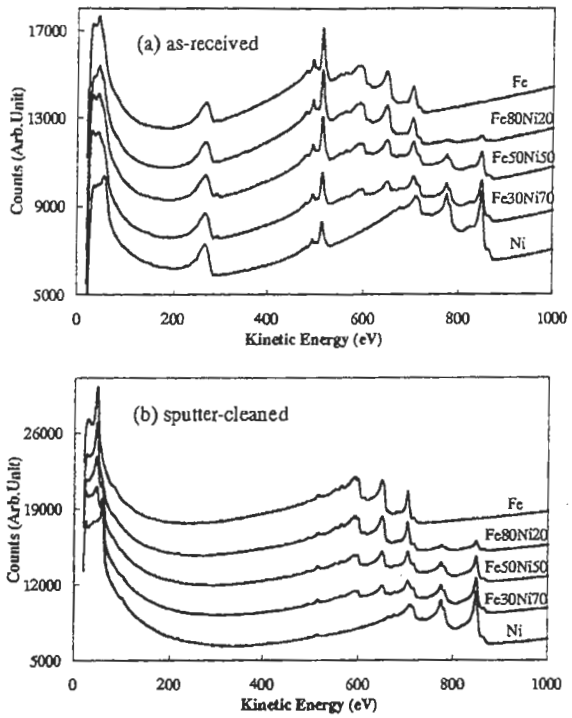

Figure 1. AES wide scan spectra of Fe, Ni and their alloys obtained from: (a) the as-received surface; (b) the sputter-cleaned surface.

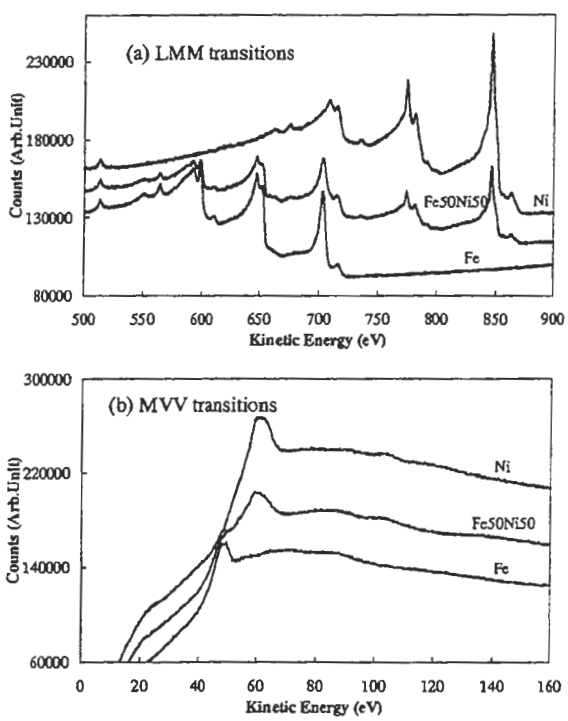

Figure 2. AES narrow scan spectra of Fe, Ni and Fe50Ni50 in the energy region of: (a) LMM; (b) MVV transitions, which are obtained from the sputter-cleaned surface with the high energy resolution of 0.05%.

same proportion. The line shapes of both LMM and MVV transitions are well reproduced by the spectral synthesis. Since the MVV transition is more sensitive to the top surface, it can be concluded that there are no significant changes on the AES spectra of Fe/Ni alloys within any depth of the sample.

3.2. REELS Measurements

Followed by the AES comparison mentioned above we measured the REELS spectrum (shown in fig. 4) of each sample, which was taken with the constant analyzer energy mode of 20eV. The energy resolution at this condition was estimated at about 1.5eV from the FWHM of the 1keV elastic peak profiles. All spectra shown in fig. 4 are normalized to the intensity of each elastic peak.

In fig. 4 two loss-peak regions are observed on the each REELS spectrum. The lower region in the energy range below 30eV is due to the interband transitions or plasmon transitions, and the higher region from 50 to 70eV is attributed to Fe and/or Ni $M_{2,3}$ core-level ionization [4,5]. Here we point out that the REELS spectrum of Fe50Ni50 in the zero-loss region between 10 and 30eV is quite similar to that of pure Ni, although it half-and-half contains Fe.

Figure 5 shows the comparison of Fe50Ni50 spectra between the measured and the synthesized. The synthesized spectrum of Fe50Ni50 was made from the pure Fe and Ni spectra shown in fig. 4 in the same proportion. Whereas the features of the measured spectrum in the core-loss region above 50eV are well reproduced by the spectral synthesis as well as AES (shown in

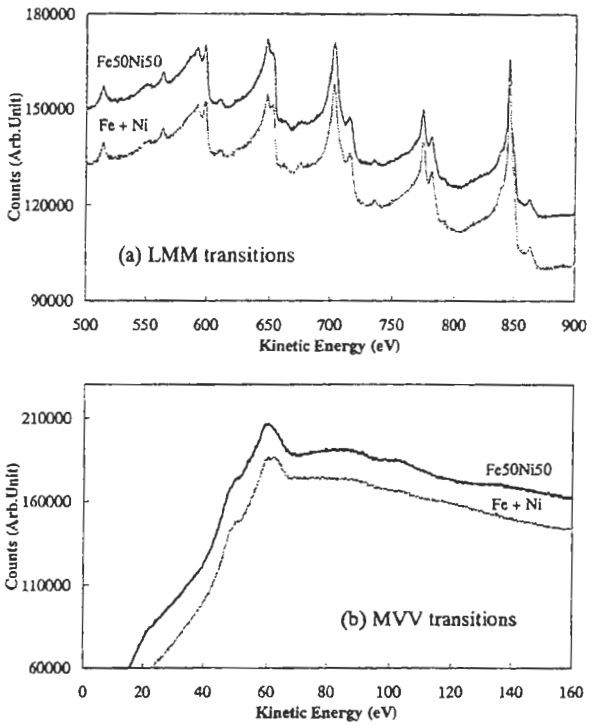

Figure 3. AES spectral comparison of Fe50Ni50 between the measured and the synthesized in the energy region of: (a) LMM; (b) MVV transitions.

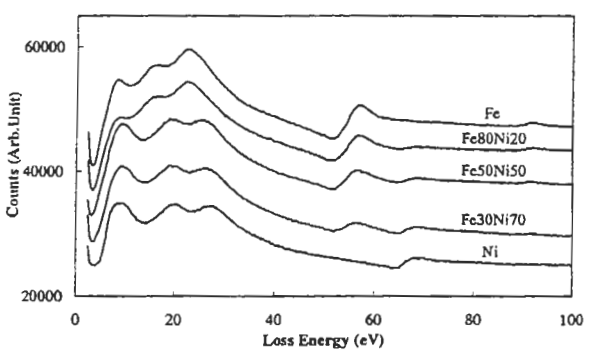

Figure 4. REELS spectra of Fe, Ni and their alloys obtained from the sputter-cleaned surface.

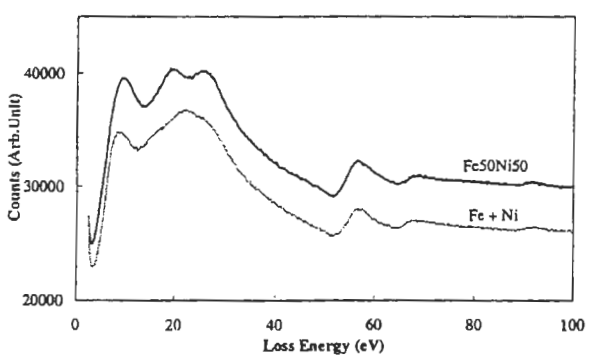

Figure 5. REELS spectral comparison of Fe50Ni50 between the measured and the synthesized.

fig. 3), in the zero-loss region they are quite different. Here we mention that it cannot be explained by the difference of the escape depth, because the kinetic energy of electrons observed in the both regions is close to 1keV. Moreover, there are no sign of the Ni segregation to the top surface according to the AES comparison mentioned above.

Since the X-ray diffraction measurements reveal that Fe50Ni50 has a fcc structure as well as pure Ni[6], we believe that the zero-loss features on the REELS spectra are strongly affected not only by the atomic composition but also by the crystal structure in the case of Fe/Ni alloys.

4. References

[1]K.Goto, J.Surf.Anal., 3, 686 (1997).

[2]N.M.Forsyth and S.Bean, Surf.Interface Anal., 22, 338 (1994).

[3]K.Wandelt and G.Ertl, Surf.Sci., 55, 403 (1976).

[4]Y.C.Lee, M.Abu-Joudeh and P.A.Montano, Surf.Sci., 143, 469 (1984).

[5]Y.C.Lee, H.Min and P.A.Montano, Surf.Sci., 166, 391 (1986).

[6]Private communications among the SASJ-MMG. Whereas Fe and Fe80Ni20 have a bcc structure, Ni, Fe30Ni70 and Fe50Ni50 have a fcc structure.